PATENT SPECIFICATION (11) 1 501 967

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(21) Application No. 30681/75 (22) Filed 22 July 1975
(31) Convention Application No. 491 075
(32) Filed 23 July 1974
(31) Convention Application No. 583 481
(32) Filed 9 June 1975 in
(33) United States of America (US)
(44) Complete Specification published 22 Feb. 1978
(51) INT CL² C09C 1/50
(52) Index at acceptance
C1A J1 J201 J243 J244 J246 J290 J300 J302 J606 J613 J614 J685 J686

(72) The inventors of this invention in the sense of being the actual devisers thereof within the meaning of Section 16 of the Patents Act, 1949, are KEITH L. HALE of 5742 Rutherglenn, Houston, Texas; WILLIAM B. CRULL, Jr., of 225 Chelsea, Dumas, Texas; NORMAN R. HIGGINS of 406 Briar Hill Drive, Houston, Texas, and DON T. NORMAN of 1418 Alice Lane, Sulphur, Louisiana; all of the United States of America and all citizens of the U.S.A.

(54) CARBON BLACK

(71) We, CONTINENTAL CARBON COMPANY, a Corporation organised under the Laws of the State of Delaware, United States of America, of Houston, Texas, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a new family of rubber-reinforcing high-structure carbon blacks having a novel combination of properties which, when used to manufacture tire tread rubber compounds, affords high treadwear resistance even at relatively high oil loadings.

A discussion of the various types and grades of carbon black is presented in "Analysis of Carbon Black," *Encyclopedia of Industrial Chemical Analysis* (1969) Vol. 8, pp. 179—243. Table 4 on pp. 198—199 lists some of the properties of various types and grades.

Since the publication of this reference, various new HAF blacks have been developed with the following typical analytical properties:

Grade (ASTM No.)	Grade (Common)	Surface Area[1]	Tint[2]	DBP[3]	24M4[4]
N—339	HAF—HS	90	111	122	103
N—347	HAF—HS	90	105	122	100
N—358	HAF—VHS	90	98	150	115

Notes:

1. Iodine No. per ASTM Test Method D—1510—70, the common industry standard test method used to classify carbon black by grade (SAF, ISAF, IISAF, HAF, etc.). Generally, the higher the iodine number, the smaller the particle size and the greater the surface area.

2. Tint (tinting strength) is another indication of particle size which has become important in evaluation of carbon black. High tint indicates small particle-size and has been associated with high rubber reinforcing properties. The test method is ASTM D—3265—73, and the reference black is IRB #3. For purposes of this invention, we use the Densichron light reflectance instrument and read paste tint through the slide on which the sample is spread.

3. Dibutyl phthalate absorption per ASTM Test Method D—2414—72. This is a measure of structure discussed on pp. 187—191 of the above reference.

4. 24M4 is the designation of a relatively new method, now well known in the art as ASTM D—2414 (tentative), of subjecting a carbon black sample to repeated compressions prior to determining the DBP absorption. The 24M4 value is indicative of the permanent or persistent (as contrasted with transient) structure of the carbon black and is therefore a more meaningful indication of structure. A suitable press and method are disclosed in U.S. patent 3,548,454, the method being described specifically in column 4, line 27—column 5, line 2.

A need has existed for a carbon black with a higher structure than N—339 but with equivalent performance (particularly treadwear resistance) properties when compounded with higher oil loading. The capability to add additional oil is particularly beneficial because this reduces the overall cost of the rubber compound due to the relatively low cost of the oil as compared with the other compound ingredients. This need presented a problem because, for a given grade of black, a significant increase in structure could not be obtained without sacrificing treadwear resistance. Also, the addition of oil has been associated with a decrease in treadwear resistance. It is the object of this invention to provide such an improved HAF—VHS black (one grade of which is hereinafter identified as X—1303) (VHS means "very high structure". It is a further object of this invention to provide a family of blacks (including X—1303) having corresponding combinations of properties.

N—358 (a very high structure HAF) does not satisfy such need because it does not have the high treadwear resistance of N—339, undoubtedly because it does not have the high tint and high 24M4 of the present blacks.

Without limiting ourselves, we offer the following explanation for our invention.

As indicated above, iodine adsorption number is often taken as an indicator of the specific surface area of the ultimate carbon black particles. The 24M4 DBP absorption is often taken as an indicator of carbon black structure, i.e., the amount of aggregation of the ultimate particles. These two tests are thus related to the size of the ultimate carbon black particles and to the size of the particle aggregates.

The size of all particles and aggregates are not uniform within a grade of carbon black but are distributed over a range of sizes. The distribution of sizes for a sample of carbon black is typically skewed to the right similar to a log-normal distribution so the modal size (peak of curve) is usually less than the arithmetic mean size. For example see Figures 3—5, pp. 184—185 of "Analysis of Carbon Black," *Encyclopedia of Industrial Chemical Analysis.*

The low reinforcing grades of furnace black are characteristically skewed far to the right resulting in a low specific particle surface area. The more reinforcing grades of carbon black approach more and more toward a symmetrical distribution resulting in an increasing specific particle surface area. The arithmetic mean diameter approaches more and more the modal diameter.

This suggests that for a given specific particle surface area (or iodine number), the more symmetrical and narrow the particle size distribution, the more the specific projected cross-sectional area (hiding power or tinting strength). Further, the more symmetrical and narrow the aggregate size distribution, the more the specific aggregate surface area (24M4 DBP absorption).

The advantage of higher tinting strength and higher 24M4 DBP of carbon black at a given Iodine No. level is higher reinforcing, better processability and higher oil and black loadings in a rubber compound.

Our invention is a new family of oil furnace carbon black, having iodine numbers (I_2) (as hereinbefore defined) within the range of 60—150, and having the following relationships between iodine number, tint (as hereinbefore defined) and persistent structure (24M4) (as hereinbefore defined):—

$$125.1-[\exp][4.83274-0.033969(I_2)]\leq 24M4\leq 140.6-[\exp][4.94743-0.03054(I_2)]$$

and

$$112.8-[\exp][4.72852-0.026274(I_2)]\leq Tint\leq 122.2-[\exp][4.80471-0.026123(I_2)]$$

No one of these properties is novel *per se*, but we believe that the above combination of properties is novel and unobvious.

Referring to the accompanying drawings,

Figure 1 is a drawing showing isopleths of carbon black and oil loadings for various Treadwear Index levels within a defined range of rubber hardness values, for a prior art black.

Figure 2 is a drawing of similar isopleths for one of the carbon blacks of this invention.

Figure 3 is a graph illustrating the mathematical formulae describing the properties of the present family of carbon blacks.

As is well known in the art, the properties of carbon black depend upon reactor (furnace) design and the operating conditions employed, assuming that the feedstock is a conventional carbon black oil of satisfactory quality.

A reactor design which we have found to be satisfactory for manufacture of our blacks has the configuration of the converging section-to-throat-to diverging section reactor described in U.S. patents 3,256,066 and 3,741,165, particularly Figure 2 of 3,256,066 and Figures 1 and 2 of 3,741,165, with a throat diameter of 5—7 inches.

Suitable operating conditions for the manufacture of our X—1303 carbon black are:

Air Rate, SCFH	130,000—200,000
Air Preheat Temp., °F.	500—700
Feedstock Oil Rate, GPH(US)	190—260
Air/Fuel Gas Ratio	12/1—17/1
Distance, Feedstock Nozzle to Throat	30—60"
Distance, Quench to Throat	4—8'
Distance, Feedstock Nozzle to Quench	8—12'
Reactor Pressure Drop Across Throat	1.0—8.0 psig
Reaction Temp., °F.	3,000—3,300
Reaction Time (Milliseconds)	8—50

The feedstock can be any carbon black oil of commercial quality. Such oils are well known in the carbon black industry and typically have the following specifications:

API Gravity	+2.0
Viscosity SFS @ 122°F.	40.1
BS&W, %	0.22
Ash, %	0.01
Asphaltenes, %	4.31
Carbon, %	90.67
Hydrogen, %	8.12
Sulfur, %	0.54
BMCI	109.5

EXAMPLES.

A number of pilot plant runs were made utilizing the reactor described above, the operating conditions described above and in TABLE I, and a feedstock comparable to that described above. Analytical properties of representative blacks of the present invention are shown in TABLE II below.

TABLE I

Air Rate, SCFH	150,000
Oil Rate, GPH(US)	200—220
Air Preheat, °F.	610
Oil Spray Pressure, psig	80—95
Air/Gas Ratio	15/1
Total reaction time, milliseconds	13—40
Reaction time, feedstock injection to throat, milliseconds	3—10

TABLE II

Run No.—	1303A$_1$	1332A$_1$	1363A	1382A	1390B	1382A	1382A	1406A	1382A	1435H
Iodine Number	91.3	91.3	89.4	93.1	91.9			90.0		88.8
N_2 Surface Area ASTM D—3037—73	99.8	103.2	103.5	103.9	101.8			95.5		107.3
DBP	150.0	149.1	144.2	148.7	151.3			162.9		153.4
24M4	121.5	124.3	120.4	121.8	128.0			119.9		122.7
Tint v. IRB #3	105	105	112	106	110.0			106		114
Density, lbs/ft^3 ASTM D—1513—60	19.7	19.1	19.9	20.4	19.4			17.0		18.8

TABLE II (Continued)

Run No.—		1303A$_1$	1332A$_1$	1363A	1382A	1390B	1382A	1382A	1406A	1382A	1435H
ASTM Recipe[1] v. IRB #—		3	3	3	3	3			3		4
	Min.										
Tensile Strength ASTM D—412—68	30	—400	—410	—350	—500	—350			—470		—510
Ultimate Elongation	30	—100	—120	—90	—120	—120			—130		—130
Modulus @ 300%	30	+550	+670	+690	+560	+550			+510		+550
Mooney @ 250°F. Min. Visc.		+29.4	+28.0	+38.6	+27.6	+37.2			+25.7		+26.7
t_1		—4.0	—1.1	—7.0	—6.2	—4.9			—1.7		—3.0
t_{35}		—4.6	—1.0	—8.2	—6.9	—5.8			—2.3		—3.8
ASTM Recipe v. IRB #—		3	3	3	3	3			3		4
	Min.										
GH Rebound, % ASTM D—1054—66	40	—2.1	—3.1	—2.8	—3.1	—2.6			—2.9		—3.9
Firestone Rebound, %	40	—1.1	—2.8	—3.5	—4.0	—1.0			—3.5		—4.0
Firestone HBU, °F. ASTM D—623—67; Load 250 lbs; oscillation 0.3 in.	40	+42	+51	+33	+56	+39			+53		+46

TABLE II (Continued)

Run No. –		1303A$_1$	1332A$_1$	1363A	1382A	1390B	1382A	1382A	1406A	1382A	1435H
NBS Recipe[2] v. IRB #—		3	3	3	3	3			3		4
	Min.										
Tensile Strength	50	–290	+290	+60	–340	–100			+240		+300
Ultimate Elongation	50	–70	–100	–90	–130	–110			–100		–140
Modulus @ 300%	25	+1320	+1060	+910	+850	+1040			+1090		+1040
Modulus @ 300%	50	+1290	+1050	+1120	+1030	+910			+1150		+1110
Mooney @ 280° F. Min. Visc.		+14.0	+12.0	+15.0	+13.0	+11.6			+9.4		+15.8
t_5		–1.2	+0.4	+1.8	–1.1	–1.6			–1.3		–1.2
t_{35}		–1.7	+0.9	–1.4	–1.6	–3.2			–2.5		–2.2
NBS Recipe v. IRB #—		3	3	3	3	3			3		4
Extrusion, gm/m. ASTM D2270—73, Method B		35.6	36.6	38.3	38.9	36.5			39.0		37.7
gm/m, IRB #3		45.5	46.0	45.9	45.0	47.5			48.0		47.7
% of IRB #3		78.2	79.6	83.0	86.4	76.8			81.3		79.0
Treadwear Recipe[3]											
Parts Black		65	65	65	65	65	63	63	65	65	65
Parts Oil		31.25	31.25	31.25	31.25	31.25	43.25	33.25	31.25	31.25	31.25
	Min.										
Tensile Strength	30	2900	3080	3080	2890	3140	2750	3100	2970	3070	3020
Ultimate Elongation	30	360	400	440	380	420	460	420	400	400	380
Modulus @ 300%	30	2230	2280	1950	2160	2100	1680	2150	2150	2160	2300
Hardness, Shore A ASTM D—2240—68	30	69	69	70	65	66	64	70	68	70	68

TABLE II (Continued)

Run No.—		1303A$_1$	1332A$_1$	1363A	1382A	1390B	1382A	1382A	1406A	1382A	1435H
Treadwear Recipe (Continued)											
Mooney @ 280°F. Min. Visc.		86.5	43.8	44.4	43.0	42.7	28.5	40.0	43.0	44.0	47.0
t_5		9.2	9.3	12.7	13.2	12.5	15.8	12.7	14.3	12.9	12.7
t_{35}		10.6	10.9	14.9	15.7	14.9	18.3	14.7	16.3	15.2	14.4
	Min.										
GH Rebound, %	40	52.2	52.5	51.6	50.8	51.0	54.8	54.9	53.4	52.9	52.3
Firestone HBU, °F. (Control; Sample)	40	265;265	270;272	253;265	254;261	260;253	265;238	265;249	277;279	271;269	261;270
Extrusion, Wt., gm/m		34.2	33.3	39.4	35.5	35.9	37.9	36.1	35.4	35.0	34.5
Extrusion, Diam., Inches		0.240	0.239	0.242	0.249	0.246	0.255	0.253	0.249	0.244	0.244
Tread Hardness, Shore A ASTM D—2240—68			70	70	69	67	63	67	70	69	69
Treadwear Index		99	99	100	94	104	103	98	97	97	97

It will be found that the values for iodine number, tint and persistent structure (24M4) of the above Runs satisfy the relationships set forth above when inserted in those relationships.

Footnotes to TABLE II

1. ASTM D—3192 73 Natural Rubber Recipe.
2. Industry synthetic recipe:

Ingredients	Parts by weight
SBR 1500	100.0
Zinc Oxide	5.0
Stearic Acid	1.50
Benzothiazyl Disulfide	2.00
Carbon Black	50.00
Sulfur	2.00

3. Treadwear recipe and procedure are described in TABLE III and description below.

TABLE III

The standard treadwear recipe for the treadwear tests is as follows:

Ingredients	Parts by Weight
SBR—1500 (rubber)	50.00
SBR—1710 (rubber + oil)	34.40
Ameripol CB—441 (rubber + oil)	34.40
"Flexone"* 3C	1.50
"Wingstay"* 100	0.50
Zinc Oxide	3.00
Stearic Acid	1.50
"Nobs"* Special	1.25
MBTS	0.10
DPG	0.50
Carbon Black Variable (Base Case 65.00)	
Petroflux LV (oil) Variable (Base Case 12.50)	

* registered Trade Marks.

Note: All of these ingredients are well known in the art and are described in "Materials and Compounding Ingredients for Rubber," *Rubber World*, Bill Communication, Inc., N.Y. 1970 Edition.

It will be noted that the amount of carbon black and the amount of Petroflux LV (a commonly used extender oil) are variable. Rubber compounders desire flexibility in compounding so that for a given desired hardness and Treadwear Index, they can use a greater amount of carbon black and/or oil loading. In the above standard treadwear recipe, the SBR 1710 and Ameripol CB—441 each contain 37.5 parts of oil per 100 parts of rubber, so that the Base Case formulation (65 parts carbon black and 12.50 parts added extender oil Petroflux LV) actually contains 31.25 parts total oil per hundred parts of rubber. In the following tables of data, therefore, the parts oil means the total oil; in other words, a treadwear recipe which contained 46.25 parts oil had 15 parts of Petroflux LV in addition to the Base Case amount of 12.50 parts of oil.

For the treadwear tests, rubber compounds were prepared from these ingredients using a Banbury mixer and accepted mixing procedures and practices. Carefully prepared batches were pressed into separate sections. Multiple sections were applied to buffed tire carcasses, retreaded and cured for sixty minutes at 302°F. (150°C). The control black was an ISAF—HM (N—220) black having an iodine number of 116—120, a Densichron Tint (v. IRB 3) of 110—112, a DBP of 115—117, and a 24M4 of 97—101. The tires were placed on automobiles and driven under carefully controlled uniform test conditions for 7200 miles. The change in tread groove depth was measured and compared to the original depth to get a tread loss for each compound. The samples were compared to the control black arbitrarily assigned a Treadwear Index value of 100.

Another series of pilot plant and plant runs conducted as above resulted in production of blacks as shown in TABLE IV. Again it will be found that the given values for iodine number, tint and persistent structure (24M4) satisfy the relationships given above.

The values quoted for Runs PCX125 are those for two separately mixed batches of rubber/black compositions and, as is well known, no two separate batches will give exactly identical results in rubber tests.

TABLE IV

Run No.—		PCX125	PCX125	1445A	1438A	1438A	PCX125	WLX114	WLX114	X1382A	BKX47	BKX47
Iodine Number		91.3		88.8	92.5		83.8			89.3	84.4	
N_2 Surface Area		107.9		105.8	104.5		104.1			101.5	96.6	
DBP		153.9		147.7	150.4		151.0			150.9	156.8	
24M4		122.9		122.5	120.8		118.8			120.3	120.0	
ASTM Dens. Tint v. IRB #3		106.8		108.3	106.0		107.6			104.2	102.4	
Density, lbs/ft^3		19.4		20.1	20.5		18.9			19.8	18.7	
ASTM D—3192—73 v. IRB #4	Min.											
Tensile Strength	30	–260		–390	–340		–350			–390	–410	
Ultimate Elong.	30	–80		–80	–110		–100			–120	–90	
Modulus @ 300%	30	+420		+380	+450		+590			+560	+610	
Mooney @ 250°F. ASTM D—1646—72 Min. Visc.		+32.4		+23.2	+16.0		+34.0			+27.0	+16.0	
t_5		–0.7		–0.9	–0.5		–3.2			–2.9	–2.9	
t_{35}		–0.8		–1.0	–0.8		–3.7			–3.1	–3.5	
ASTM D—3192—73 v. IRB #4	Min.											
GH Rebound, %	40	–0.8		–3.2	–2.6		–0.9			–1.0	–3.0	
Firestone Rebound, %	40	–1.5		–4.2	–1.0		–2.6			–2.5	–1.2	
Firestone HBU, °F.	40	+37		+41	+52		+25			+40	+25	

TABLE IV (Continued)

Run No.—		PCX125	PCX125	1445A	1438A	1438A	PCX125	WLX114	WLX114	X1382A	BKX47	BKX47
ASTM Synthetic Rubber Recipe D—3191 v. IRB #4	Min.											
Tensile Strength	50	—60		—130	—10		—410			+20	—340	
Ultimate Elong.	50	—100		—60	—70		—110			—50	—120	
Modulus @ 300%	35	+980		+880	+870		+710			+740	+1060	
Modulus @ 300%	50	+980		+950	+840		+890			+830	+1050	
Mooney @ 280°F. Min. Visc.		+21.7		+17.5	+19.3		+16.5			+17.0	+18.0	
t_5		—2.0		—1.0	—3.4		—5.2			—5.3	—6.3	
t_{35}		—3.2		—1.8	—4.7		—5.9			—5.8	—7.4	
Extrusion weight gm/m		37.1		37.4	36.		38.5			36.8	36.6	
gm/m, IRB #4		48.0		48.3	48.		48.4			50.2	48.4	
% of IRB #4		77.3		77.4	75.		79.5			73.3	75.6	

TABLE IV (Continued)

Run No.—		PCX125	PCX125	1445A	1438A	1438A	PCX125	WLX114	WLX114	X1382A	BKX47	BKX47
Treadwear Recipe												
Parts Black		65	65	65	65	65	65	65	65	65	65	65
Parts Oil		31.25	31.25	31.25	31.25	46.25	46.25	31.25	46.25	31.25	31.25	46.25
	Min.											
Tensile Strength	30	2920	3040	3060	3000	2270	2890	3240	2780	2960	3080	2700
Ultimate Elong.	30	360	390	410	380	430	470	390	450	350	360	460
Modulus @ 300%	30	2400	2200	2140	2260	1400	1650	2450	1680	2480	2520	1650
Hardness, Shore A		67	70	68	69	61	60	68	60	68	65	63
Mooney @ 280°F. Min Visc.		48.0	49.0	46.2	46.2	31.2	32.0	47.0	35.0	44.5	44.5	32.0
t_5		13.2	13.2	14.5	13.9	16.2	17.4	14.4	18.8	14.6	13.4	15.0
t_{25}		15.2	15.0	16.7	16.0	18.2	19.6	16.6	21.2	16.7	15.6	17.0
GH Rebound, %	40	54.8	54.8	53.6	53.0	51.6	56.4	55.8	54.2	56.2	55.6	53.7
Firestone HBU, °F.												
Control	40	250	266	266	266	266	260	260	260	252	252	253
Sample	40	252	261	262	270	260	252	259	248	257	246	243
Extrusion, Wt., gm/m		34.5	34.8	34.7	32.8	35.6	38.0	35.5	38.4	33.2	32.6	36.0
Extrusion, Diam., In.		0.250	0.242	0.243	0.232	0.250	0.258	0.251	0.260	0.240	0.260	0.250
Tread Hardness		69	69	68	69	61	63	70	62	69	69	65
Treadwear Index		96	94	100	94	103	97	96	97	98	95	101

The above data are representative of data which we have obtained showing that we have produced a very high-structure (approximately 150 DBP) HAF-grade black having treadwear properties (Treadwear Index about 95—104) closely approximating our control black in our base case standard treadwear recipe. Previous data obtained from testing of prior HAF blacks showed that as the DBP was increased from about 116 to 147 the Treadwear Index dropped from about 103 to about 90. Typical Treadwear Index values for prior art N—339, N—347, and N—358 blacks are 100, 93 and 91 respectively.

TABLE V shows the results of additional studies of varying the oil/black loading in our standard treadwear recipe described above, using samples of black from Run No. 1382A. The control black was the ISAF—HM (N—220) control black described above.

TABLE V

Sample No.		Control	247 B9316	247 C9316	247 D9316	254 B9316	247 E9316	254 C9316	247 F9316	247 G9316	247 H9316	254 D9316	254 E9316
Batch No.		15	1	2	3	—	4	—	5	6	7	—	—
Parts Black		65	53	63	63	63	63	63	73	73	73	53	63
Parts Oil		31.25	43.25	53.25	43.25	43.25	33.25	33.25	63.25	53.25	43.25	23.25	23.25
Treadwear Index		100	94	95	101	103	106	98	93	104	100	95	93
	Min.												
Tensile Strength	30	2980	2310	2400	2570	2750	2760	3100	2310	2420	2670	3280	3400
Ultimate Elong.	30	480	490	540	470	460	400	420	530	460	400	410	370
Modulus @ 300%	30	1530	1150	1170	1510	1680	1980	2150	1180	1450	1920	2180	2660
Hardness, Shore A	30	65	56	57	62	64	65	70	60	60	67	66	69
Mooney @ 280°F. Min. Visc.		38.3	28.0	24.2	30.0	28.5	42.0	40.0	24.2	30.0	40.0	41.0	53.0
t_5		18.5	18.0	21.0	16.0	15.8	13.6	12.7	16.8	16.9	13.6	14.8	12.0
t_{35}		21.3	20.4	23.8	18.6	18.3	15.7	14.7	19.7	19.4	16.2	17.6	14.0

TABLE V (Continued)

Sample No.—	Min.	Control	247 B9316	247 C9316	247 D9316	254 B9316	247 E9316	254 C9316	247 F9316	247 G9316	247 H9316	254 D9316	254 E9416
GH Rebound	40	49.6	52.1	48.2	49.9	54.8	52.0	54.9	45.4	45.9	47.7	58.4	55.6
Firestone HBU	40	243	232	261	254	238	261	249	268	269	281	231	257
Extrusion, Wt., gm/m		45.2	47.3	41.8	38.4	37.9	36.3	36.1	38.0	35.4	32.8	39.4	35.9
Extrusion, Diam., In.		0.278	0.286	0.267	0.255	0.255	0.248	0.253	0.254	0.244	0.236	0.265	0.250
Initial Hardness		68	58	59	64	63	68	67	60	65	69	67	71

Figures 1 and 2 are isopleth drawings showing the results of carbon black/oil loading studies on a prior art black (N—339) and on the blacks, of the present invention, respectively. The Treadwear Index values (represented by the curved lines) were derived as described above and were compared with the control black in the standard recipe (TABLE III). The Shore A Hardness (per ASTM Test Method 2240—68) is a standard industry property of cured rubber stock. "Carbon Black Loading phr" means the parts of carbon black per hundred parts of rubber used to prepare the overall composition. "Total Oil Loading phr" means the total parts of extender oil (commonly used in rubber compounding and listed as Petroflux LV in the recipe on TABLE IV) per hundred parts of rubber used. These isopleths show that our black will afford to a rubber compounder greater flexibility, in that for a given desired hardness and Treadwear Index, he can use a greater amount of carbon black and/or total oil loading.

The relationships shown in Figures 1 and 2 were surprising in that the present blacks, although having a lower tint and therefore a lower expected Treadwear Index than N—339, actually turned out to have higher Treadwear Index values through a broad range of oil/black loadings.

Referring to Figures 1 and 2, it can be seen that when using N—339, and a carbon black loading of 85 phr, the maximum oil loading, for a Treadwear Index of 93 and a Shore A hardness of 65, was 55 phr of oil. On the other hand, using the present blacks at the same black loading of 85 and compounding to the same hardness of 65, would allow an oil loading of 70 phr for a Treadwear Index of 94.

TABLE VI shows the "pound-volume" cost, along with other important physical properties, for N—339 and one of the present blacks identified as X—1303 compounded as per TABLE III. The compound cost is the dollar cost per pound multiplied by the compound specific gravity, as explained in *The Vanderbilt Rubber Handbook*, R. T. Vanderbilt Company, Inc., N.Y., N.Y., 1968, pp. 596597. The ingredient cost was the prevailing F.O.B. price for September, 1974.

TABLE VI

Black	Parts Black	Parts Oil	Treadwear Index	Compound Cost
N—339	85	55	93	0.2425
X—1303	85	70	94	0.2293

Those skilled in the art will note that the blacks in TABLES II—VI are in the N—300 series having iodine numbers in the range of about 84—93. Additional pilot plant runs were made to produce additional blacks of different iodine numbers (about 110—125 and 65—75, respectively) and a combination of high tints and high permanent or persistent structures. The results of these runs are shown in TABLE VII. Again it will be found that the given values for iodine number, tint, and persistent structure (24M4) satisfy the relationships given above.

TABLE VII

Run No.—	1498A	1498B	1055A	1513C	1503A	1503D	1507A	1509A	PCX144A
Air Rate, 1000 SCFH	250	250.2	248	249.4	244.3	244.5	250	248.8	150
Oil Rate, GPH(US)	315.9	316.9	311.6	302.2	458	461.5	475.4	477.2	271
Air Preheat, °F.	←----------610----------→								730
Oil Spray Pressure, psig	340	335	310	315	195	200	210	125	110
Air/gas ratio	←----------15----------→								17.5
Total reaction time, Milliseconds	←----8—30----→				←----23—50----→				
Reaction time, feedstock to throat, milliseconds	←----3—10----→				←----3—10----→				
Iodine Number	116.9	113.0	111.0	121.3	65.6	65.6	68.8	66.3	71.2
N_2 Surface Area	124.3	123.5	117.5	134.4	77.1	74.8	79.4	77.3	84.5
DBP	175.5	175.2	156.9	142.0	153.1	159.0	154.6	164.3	162.2
24M4	133.8	135.0	125.0	125.1	112.5	115.9	114.5	115.4	118.5

TABLE VII (Continued)

Run No.—	1498A	1498B	1055A	1513C	1503A	1503D	1507A	1509A	PCX144A
Tint v. IRB #3	110.0	109.6	110.7	114.5	94.0	92.7	97.6	94.4	95.6
Density, lbs/ft³	15.5	17.5	17.4	19.8	18.7	18.0	18.6	17.9	17.8
Grade of Black	ISAF—VHS	ISAF VHS	ISAF—VHS	ISAF—VHS	T—VHS	T—VHS	T—VHS	T—VHS	T—VHS

TABLE VIII gives rubber test data on samples of the new improved blacks made in Runs 1513C and PCX144A.

TABLE VIII

Run No.		1513C (ISAF—VHS)	PCX144A (T—VHS)
ASTM D—3192—73 v. IRB #4	Min.		
Tensile Strength	30	—380	—860
Ultimate Elongation	30	—70	—160
Modulus @ 300%	30	+250	+550
Mooney, ML 1+4 @ 212°F.		+34	+20
GH Rebound, %	40	—5.3	—1.1
Firestone Rebound, %	40	—4.0	—1.0
Firestone HBU, °F.	40	+45	+27

TABLE VIII (Continued)

Run No.		1513C (ISAF—VHS)	PCX144A (T—VHS)
ASTM Synthetic Rubber Recipe D—3191 y, IRB #4	Min.		
Tensile Strength	50	—60	—490
Ultimate Elongation	50	—80	—120
Modulus @ 300%	35	+380	+850
Modulus @ 300%	50	+620	+800
Mooney, ML 1+4 @ 212°F. ASTM D—1646—72		+29	+20
Extrusion gm/m, IRB #4		—11.5	—13.1
% of IRB #4		76.7	73.5
Treadwear Recipe	Min.		
Parts Black		65	65
Parts Oil		46.25	41.25
Tensile Strength	30	2620	2890
Ultimate Elongation	30	560	480
Modulus @ 300%	30	1360	1310
Hardness, Shore A		55	58
Mooney, ML 1+4 @ 212°F.		51	49
GH Rebound, %	40	41.2	48.5
Firestone HBU, °F. (Control; sample)	40	275	265;258
Extrusion, Wt., gm/m		40	35.1
Extrusion, Diam., Inches		.266	.250
Tread Hardness		62	62
Treadwear Index		99	92

Note: The control was the same as the control of TABLE V.

The above data (TABLES VII and VIII) show that we have developed a new ISAF—VHS black which, when compounded with 14 additional parts of oil (46.25 parts total oil compared with 31.25 in our standard treadwear recipe), resulted in a Treadwear Index of 99. This compares favorably with the Treadwear Index of 100 with our control black at standard oil loading. This results from the combination of higher 24M4 and high tint.

The above data (TABLES VII and VIII) also show that we have developed a new T—VHS black which, when compounded with 10 additional parts of oil (41.25 total oil compared with 31.25 in our standard treadwear recipe), resulted in a Treadwear Index of 92. This compares favorably with a Treadwear Index of about

86—89 (at standard oil loading of 31.25 parts) for the closest prior art black T—HS (N—351). Again, this results from a higher 24M4 combined with a high tint as shown by the following typical values:

	24M4	Tint v. IRB #3	Treadwear Index (10 Additional Parts Oil)
T—HS (N—351)	98	103	87
Our new T—VHS	115	95	92

WHAT WE CLAIM IS:—

1. A family of grades of oil furnace carbon black, having iodine numbers (I_2) (as hereinbefore defined) within the range of 60—150, and having the following relationships between iodine number, tint (as hereinbefore defined), and persistent structure (24M4) (as hereinbefore defined):

$$125.1-[\exp][4.83274-0.033969(I_2)] \leq 24M4 \leq 140.6-[\exp][4.94743-0.03054(I_2)]$$

and

$$112.8-[\exp][4.72852-0.026274(I_2)] \leq Tint \leq 122.2-[\exp][4.80471-0.026123(I_2)]$$

2. The carbon black of Claim 1 in which the iodine number falls within the range of 84—93.

3. The carbon black of Claim 1 in which the iodine number falls within the range of 110—125.

4. The carbon black of Claim 1 in which the iodine number falls within the range of 65—75.

5. An oil furnace carbon black in accordance with Claim 1 substantially as hereinbefore described with reference to any one of the foregoing Tables II, IV and VII.

6. A method of making an oil furnace carbon black in accordance with Claim 1 substantially as hereinbefore described.

TREGEAR, THIEMANN & BLEACH,
Chartered Patent Agents,
Enterprise House,
Isambard Brunel Road,
Portsmouth PO1 2AN,
and
49/51, Bedford Row, London, WC1V 6RU.
Agents for the Applicants.

Printed for Her Majesty's Stationery Office by the Courier Press, Leamington Spa, 1978.
Published by the Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.

1501967 COMPLETE SPECIFICATION
2 SHEETS *This drawing is a reproduction of the Original on a reduced scale*
Sheet 1

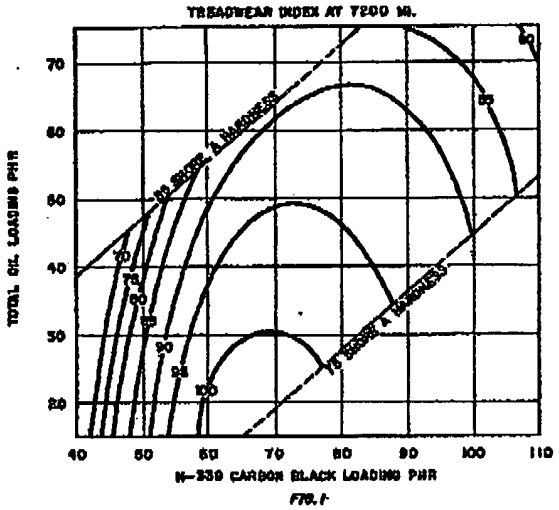

FIG. 1

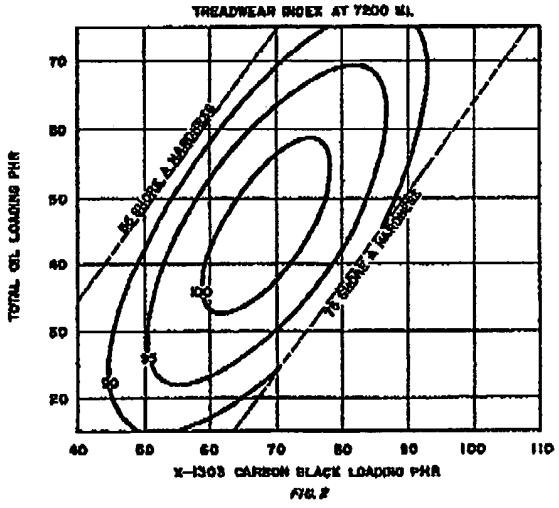

FIG. 2

1501967 COMPLETE SPECIFICATION
2 SHEETS *This drawing is a reproduction of the Original on a reduced scale*
Sheet 2

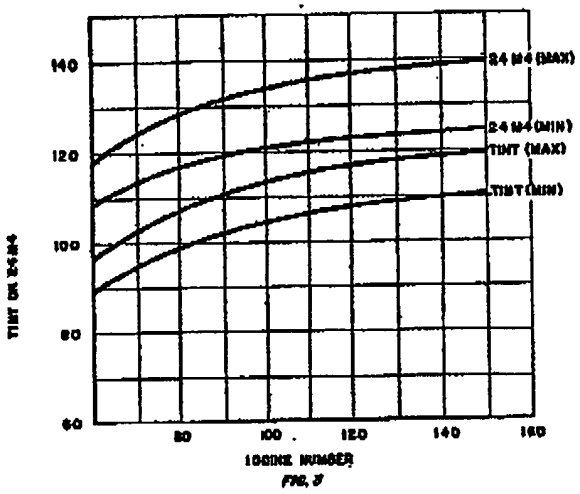

FIG. 2